

The Sorption Properties of Exploding Wire Prepared Ag, Fe and ZnO Nanoparticles for Nitrite Removal from Water

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Abstract

Part of the research work carried out at our university is presented. Ag, Fe and ZnO nanoparticles are prepared by the lab made electro-exploding wire system. The prepared nanoparticles are utilized in water pollution treatment. Laboratory experiments have been designed for exploring the sorption properties of these nanoparticles for the removal of nitrite from water. Spectrophotometric method was used for the determination of nitrite in the concentration range of 0.05–2.5 mg/L. At fixed concentrations of nitrite and nanoparticles (2.5:1.6) mg/L (nitrite: nanoparticles), It was observed that Ag nanoparticles possess highest sorption capability for removing nitrite followed by Fe nanoparticles. The calculated equilibrium adsorption capacities (q_e) were 980, 603 and 376 mg/g (adsorbate/adsorbent) for the nitrite sorption onto Ag, Fe and ZnO nanoparticles, respectively. The adsorption kinetic data were analyzed using the pseudo first-order, pseudo second-order and intraparticle diffusion model. Ag, Fe and ZnO nanoparticles exhibited excellent efficiencies for removing nitrite from water.

Keywords

Exploding Wire; Nanoparticles; Nitrite Sorption; Water Treatment

Introduction

Most nitrogenous materials in natural waters tend to be converted to nitrate, so all sources of combined nitrogen, particularly organic nitrogen and ammonia, should be considered as potential nitrate sources. Primary sources of organic nitrates include human sewage and livestock manure, especially from feedlots. The primary inorganic nitrates which may contaminate drinking water are potassium nitrate and ammonium nitrate both of which are widely used as fertilizers.

Nitrates themselves are relatively nontoxic. However, when swallowed, they are converted to nitrites that can react with hemoglobin in the blood, oxidizing its

divalent iron to the trivalent form and creating met-hemoglobin. This met-hemoglobin cannot bind oxygen, which decreases the capacity of the blood to transport oxygen so less oxygen is transported from the lungs to the body tissues, thus causing a condition known as met-hemoglobinemia. Met-hemoglobinemia occurs mostly in children and is also known as “blue-baby syndrome” (Shuval and Gruener 1977; Kapoor and Viraraghavan 1997).

Consequently, the concentration of nitrate and nitrite in drinking water has been regulated. The levels in drinking water of nitrate and nitrite established by the European Union legislation are 50 and 0.5 mg/L, respectively (Council Directive 1991). In the United States, EPA (2003) establishes the maximum contaminant levels of 10 mg/L for nitrate and 1.0 mg/L for nitrite in public water supplies.

Nitrate-contaminated waters are commonly treated by ion exchange or reverse osmosis. However, these traditional treatments are relatively expensive, because they require frequent regeneration of the media, or generate secondary brine wastes that may pose a disposal problem. Biological denitrification (Kim, Seagren, and Davis 2003; Davis 2007) is another alternative to remove nitrate from water because the microbial process reduces the nitrate to innocuous nitrogen gas rather than ammonium and generally results in lower operating costs as compared to ion exchange or reverse osmosis. However, this process can produce excessive biomass and soluble microbial products that require further treatment of the denitrified water (aeration and disinfection). Moreover, the denitrification process is generally slow and sometimes incomplete as compared to chemical reduction. The catalytic hydrogenation of nitrate (Horold et al. 1993; Gavagnin et al. 2002) is suitable for fast treatment of the nitrate-contaminated water but still requires an addition of a reducing agent (such as

hydrogen). In recent years, the success of iron metal in treating groundwater containing chlorinated solvents has stimulated a significant interest in the application of zero-valent iron (Maroto et al. 2009; Suzuki et al. 2012). However, nitrate reduction by Fe^0 is relatively sensitive to the solution pH, and nitrate is well known as an oxidizing inhibitor to iron corrosion due to the formation of an overlying oxide layer.

Sorption, in general, is the process of collecting soluble substances that are in solution on a suitable interface. The most efficient and commonly used adsorbent is activated carbon (Lee, Low, and Chung 1997; Wang et al. 2006). However, Activated carbon is very expensive adsorbent for removal of pollutant therefore other adsorbent must be investigated.

Nanoparticles (NP) could be an additional alternative option as a metal contaminant sorbent. Larger surface area may translate into greater sorption capacities allowing for smaller sorbent volumes and less waste for disposal. As particle size decreases more unsaturated surface atoms are exposed prompting greater reactivity. For example, nanoscale iron particles were employed for the denitrification of unbuffered nitrate solutions at initial neutral pH (Liou et al. 2005).

We have been working with the preparation of pure metallic Ag and Fe NP using a novel electro-exploding wire (EEW) technique (Sen et al. 2003; 2004; 2007). These NP exhibited interesting optical and magnetic properties (Alqudami and Annapoorni 2005, 2007; Alqudami et al. 2007, 2008; Sing, Alqudami, and Annapoorni 2010). The EEW technique is a direct & simple physical process and does not involve any chemical agent. Recent investigation showed that Ag and Fe NP prepared by EEW technique were effective adsorbents for the removal of Pb(II) and Cd(II) ions from water with high adsorption capacities at equilibrium (Alqudami, Alhemiary, and Munassar 2011).

This work aimed at investigating the adsorption behavior of Fe, Ag, and ZnO NP for the removal of nitrite from water. For this purpose, spherical Ag, Fe, and ZnO NP were prepared using EEW technique. The structural properties of these NP are published elsewhere (Alqudami and Annapoorni 2005, 2007; Alqudami et al. 2007, 2008; Sing, Alqudami, and Annapoorni 2010; Alqudami, Alhemiary, and Munassar 2011). The nitrite removal from water is investigated as functions of NP type and contact time. The adsorption properties are studied from kinetic standpoint.

Experimental

Nanoparticles Preparations

Prior to the performing experiments, Ag, Fe, and Zn wires (dia: 0.25 mm; Purity: 99.998%; Alfa Aesar) and plates (dim: 20, 20, 1 mm; Purity: 99.998%; Alfa Aesar) were cleaned using emery paper and followed by Acetone. The wires each of 20 mm length were exploded on plates of the same material type in distilled water using the EEW set-up sketched in Fig. 1. These wires have been exploded by bringing the metal wire into sudden contact with the metal plate. The above mentioned length of wire is standing for several sparks and is being replaced after several contacts. Both the electrodes (wire and plate) are subjected to a potential difference of 36 V DC supplied from three 12 V batteries connected in series. The resulted NP remain suspended in water (colloidal form). Independent experiments were performed to prepare each type of the NP. These NP were used to investigate their adsorption properties for the nitrite removal experiments.

The EEW process is very energy intensive since only relatively low voltages are applied, and also results in large quantities of NP being produced from both the consumed electrodes.

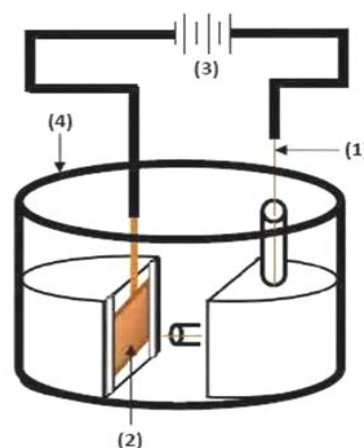


FIG. 1 A SCHEMATIC DIAGRAM OF THE ELECTRO-EXPLODING WIRE (EEW) SET UP; (1) THIN METAL WIRE, (2) METAL PLATE, (3) 36 V BATTERIES AND (4) GLASS VESSEL

Nitrite Ions Preparation

Chemicals used were of analytical grade and double distilled water was used throughout the experiments. Nitrite standard solution was prepared by dissolving 1.50 g of dried NaNO_2 in distilled water and diluting to the mark in a one-liter volumetric flask. Working solutions were prepared by appropriate dilution of the stock solution with water. A 0.04 M sulfanilic acid

solution was prepared by dissolving 1.7319 g of sulfanilic acid in water and diluting to the mark in a 250-mL volumetric flask. Acetic acid solution (6 M) was prepared by appropriate dilution of glacial acetic acid with water. A 0.5 M N,N-dimethylaniline solution was prepared by dissolving 6.3 mL N,N-dimethylaniline in water containing 20 mL of glacial acetic acid and diluting to 100 mL with distilled water. This solution was stored in a dark bottle in a refrigerator.

Determination of Nitrite Ions

A simple, rapid and sensitive spectrophotometric method was used for the trace determination of nitrite in water (Afkhani and Mogharnesband 1997; Aydin, Ercan, and Tascioglu 2005; Nagaraja, Prakash, and Bhaskara 2006).

All measurements were performed using 10-mL standard flask, containing 0.5-25 μg of nitrite along with 0.6 mL of 0.5 M N,N-dimethylaniline and dilute to the mark with water. The solutions were shaken on a rotary shaker for fixed periods and then were centrifuged at speeds of 6000 rpm for 5 minutes to remove the precipitated solid and then filtered using filter papers. 3 mL from the solution were transferred into quartz cells for measuring the absorbance at 510 nm using a Shimadzu model UV-120-01 spectrophotometer. The absorbance at 510 nm are measured in 1.00 cm quartz cells against reagent blank which is prepared in the same manner but in the absence of nitrite.

Under the optimum conditions, a calibration graph is plotted for nitrite as in Fig. 2. A straight line has been obtained in the concentration range of 0.05 – 2.5 mg/L, with a correlation of 0.99 and a regression equation of,

$$A = 0.07199 + 0.35402 C$$

Where C is the concentration of nitrite in mg/L

Kinetics of the Nitrite Ions Sorption

All measurements were performed using 10-mL standard flask, containing 25 μg of nitrite and 16 μg of nanoparticles along with 0.6 mL of 0.5 M N,N-dimethylaniline and dilute to the mark with water.

The 10-mL solutions (nitrite + nanoparticles + methyl orange in acidic media) with fixed concentrations of nitrite and nanoparticles (2.5:1.6) mg/L (nitrite: nanoparticles) were shaken on a rotary shaker for different periods, namely for 5, 10, 20, 30 and 60 minutes. Then, the solutions were centrifuged at speeds of 6000 rpm for 5 minutes to remove the

precipitated solid and filtered using filter papers. 3 mL from the decanted solutions were transferred into quartz cells for measuring the absorbance at 510 nm during the first 5 minutes by using a spectrophotometer. The concentration of nitrite ions were calculated as a function of nanoparticle type and contact time. The sorbed amount was calculated as the difference between the nitrite contents of the final solutions and control samples in the absence of nanoparticles. All experiments were performed at room temperature (30 °C).

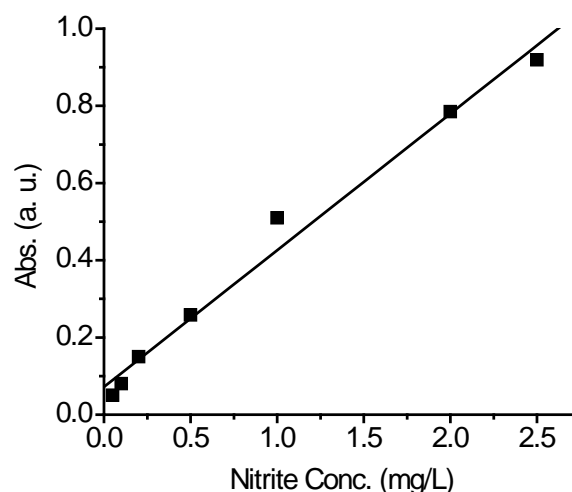


FIG. 2 A CALIBRATION GRAPH FOR NITRITE ABSORBANCE VERSUS CONCENTRATION

Results and Discussion

Effect of NP Type and Contact Time

The removal of nitrite by several adsorbents namely Ag, Fe, and ZnO NP are shown in Fig. 3 & Fig. 4.

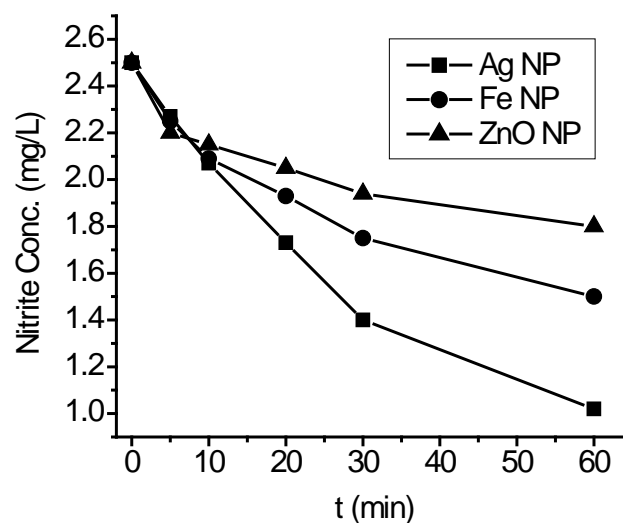


FIG. 3 ADSORPTION OF NITRITE BY Ag, Fe, AND ZnO NP AS A FUNCTION OF CONTACT TIME IN MINUTES

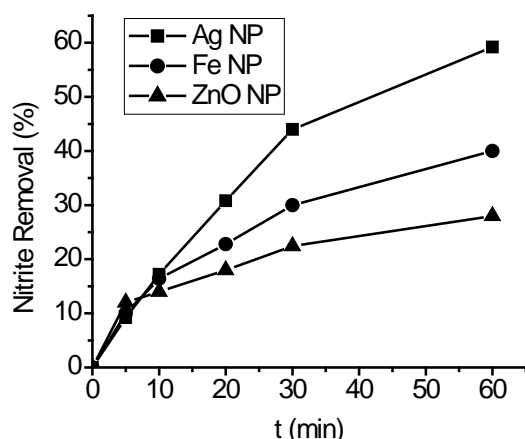


FIG. 4 REMOVAL OF NITRITE BY Ag, Fe, AND ZnO NP AS A FUNCTION OF CONTACT TIME IN MINUTES

Although NP concentrations were about one half of the nitrite concentration, the NP show remarkable removal efficiencies. 60% of the nitrite ions were removed in 60 minutes of contact with Ag NP. The removal efficiency was less by using Fe and ZnO NP at the same time of contact. The removal of nitrite was rapid in the initial stages of contact time and slowly increased with lapse of time. The variation of removal efficiency might be due to the formation of oxide shell around Fe NP and due to full oxidation of Zn NP (will be discussed in more detail on page 34).

Adsorption Kinetics

Many mathematical models are reported in the literature, all attempting to describe quantitatively the kinetic behavior during the adsorption process. Among these, the pseudo-first-order (Lagergren 1898; Ho 2004) and pseudo-second-order (Ho et al. 2000) models were used to test kinetic experimental data;

$$\frac{dq_t}{dt} = k_1 (q_t - q_e) \quad (1)$$

$$\frac{dq_t}{dt} = k_2 (q_t - q_e)^2 \quad (2)$$

where, q_e and q_t are the amounts of nitrite adsorbed (mg/g) per unit weight of adsorbent at equilibrium and at time t (min), respectively, k_1 and k_2 are reaction rate constants. The following linearised time dependent functions were obtained by integrating and rearranging Eq. 1 and Eq. 2 with the boundary conditions of $t = 0$ and $q_t = 0$ to $t = t$ and $q_t = q_e$ to yield;

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

Plots of $\log(q_e - q_t)$ versus t and of t/q_t versus t are presented in Fig. 5 and Fig. 6, respectively.

Values of k_1 , k_2 and q_e were calculated from the intercept and slope of the plots of $\log(q_e - q_t)$ vs. t , and t/q_t vs. t , and all are listed in Table 1. The results showed that the pseudo-first-order rate expression was valid for the adsorption of nitrite onto Ag NP and Fe NP. The experimental q_e values are in agreement with the calculated ones, obtained from the linear plots (Table 1) and the correlation coefficient values are higher when compared to those obtained using the pseudo-second-order rate expression for the same adsorbents. However, the adsorption of nitrite using ZnO NP showed to follow the pseudo-second-order rate equation due to relatively high value of correlation coefficient (Table 1).

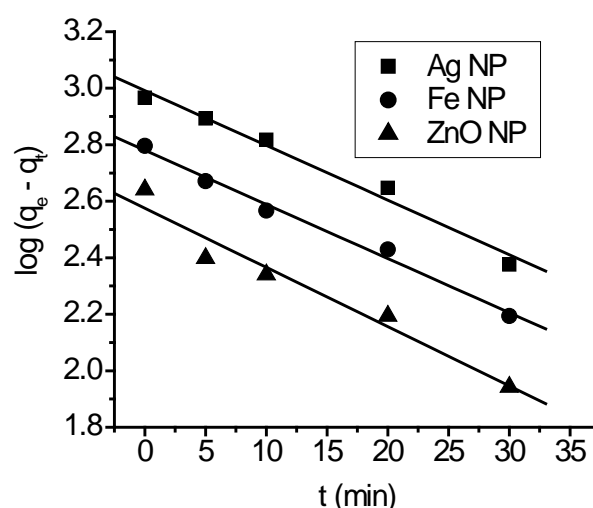


FIG. 5 APPLICATION OF THE PSEUDO FIRST-ORDER KINETIC MODEL TO THE ADSORPTION OF NITRITE

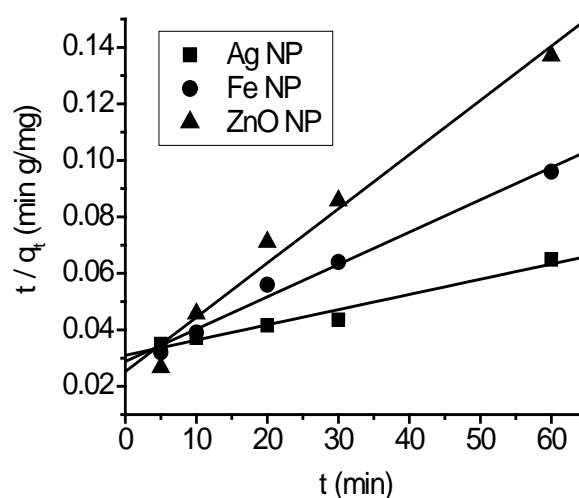


FIG. 6 APPLICATION OF THE PSEUDO SECOND-ORDER KINETIC MODEL TO THE ADSORPTION OF NITRITE

TABLE 1 NITRITE ADSORPTION KINETICS

Adsorbent NP	q_e (Exp) (mg/g)	Pseudo First-Order			Pseudo Second-Order			Intraparticle Diffusion	
		q_e (mg/g)	k_1 (min ⁻¹)	R^2	q_e (mg/g)	k_2 (g/mg min)	R^2	k_i (mg/g min ^{1/2})	R^2
Ag	925	980	0.0446	0.9817	1856	9.37×10^{-6}	0.9691	114	0.9867
Fe	625	603	0.0441	0.9905	877	45.2×10^{-6}	0.9883	81.3	0.9927
ZnO	438	376	0.0483	0.9562	520	147×10^{-6}	0.9799	61.4	0.9911

Intraparticle Diffusion Model

Adsorption is a multi-step process involving transport of the sorbate from the bulk solution to the surface of the sorbent, followed by intraparticle or pore diffusion, where sorbate move into the interior of sorbent particles, and adsorption on the interior sites of the sorbent (Bhattacharyya and Sharma 2004). The intraparticle diffusion equation can be described as:

$$q_t = k_i t^{1/2} + c \quad (5)$$

where k_i is the intraparticle diffusion rate constant (mg/g min^{1/2}). The k_i is the slope of straight line portions of the plot of q_t vs. $t^{1/2}$.

According to this model, a plot of q_t versus $t^{1/2}$ should be linear if intraparticle diffusion is involved in the adsorption process and if the plot passes through the origin then intraparticle diffusion is the sole rate-limiting step (Ozcan, Ozcan S, and Gok 2007). It has also been suggested that in instances when q_t versus $t^{1/2}$ is multilinear, two or more steps govern the adsorption process (Unuabonah, Adebawale, and Olu-Owolabi 2007; Wu, Tseng, and Juang 2009).

Fig. 7 shows the plots of q_t vs. $t^{1/2}$ for the adsorption of nitrite onto Ag, Fe and ZnO NP.

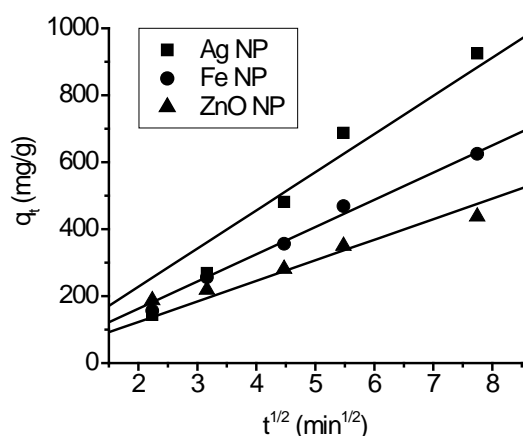


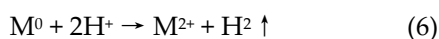
FIG. 7 INTRAPARTICLE DIFFUSION MODEL FOR THE SORPTION OF NITRITE ONTO Ag, Fe, AND ZnO NP

Given the linear plots of q_t vs. $t^{1/2}$ for the adsorption of nitrite on Ag, Fe and ZnO NP, this suggests that intraparticle diffusions are involved in the adsorption processes. The intraparticle diffusion rate constants k_i were calculated from the slopes of the straight lines and are shown in Table 1 along with the correlation coefficients. As the plots did not pass through the origin, intraparticle diffusions were not the only rate-limiting step. The values of the intercept c provide information related to the thickness of the boundary layer (Kavitha and Namasivayam 2007). Larger intercepts suggest that surface diffusion has a larger role as the rate-limiting step.

Further Discussion

To understand the sorption properties of the EEW prepared Ag, Fe, and ZnO NP, it is important to discuss the processes of NP formation using EEW technique. During NP production by under-water wire explosions, water decomposition (e.g. electrolysis) may occur. This would result in the generation of gaseous hydrogen and oxygen. Hydrogen and oxygen start to interact with the newly prepared NP. Since hydrogen (molecular or atomic forms) does not adsorb on Ag, Fe and Zn particle surfaces at room temperature (Dus and Nowicka 2003) and also is not significantly dissolved in water, it is ultimately removed to the gas phase. However, oxygen (especially atomic) could adsorb and react with the Ag, Fe and Zn surfaces at room temperature. It is also expected that during the EEW process, electrons are injected from the cathode to the NP, additionally saturated by atomic oxygen. Hence, Ag as well as Fe clusters could easily create hydrogen bonds with water particles in water environments. Finally, negatively charged Ag and Fe NP are created in the water medium due to the formation of thin oxides/hydroxyl layer around the spherical metallic core (Alqudami, Alhemiary, and Munassar 2011). Earlier investigation showed that the presence of dissolved oxygen in water is playing a key role in the

complete conversion of Zn to ZnO phase (Singh, Alqudami, and Annapoorni 2010). It is likely that the dissolved oxygen molecules are diffusing into the Zn particle slowly and reacting with the Zn atoms to form ZnO nanocrystals. Here, it is interesting to note that metal-to-metal oxide seems to depend on the properties like reactivity to oxygen and melting point. During the adsorption experiments in acidic media, it was believed that acid dissolves the oxide passive layers at low pH, and as a result, the regenerated metallic core or active metallic sites are exposed to nitrite ions. Metallic core surface (M^0) then will effectively adsorb and reduce NO_2^- . Regarding the electron transfer from the M^0 core to NO_2^- , two mechanisms have been proposed. The first proposed mechanism involves the direct electron transfer from the M^0 core to the NO_2^- , whereas the second mechanism involves indirect electron transfer via H_2 gas produced according to Eq. 6. However, there is still controversy about the dominant electron transfer process.



Considering that NO_2^- is a strong oxidant and that surface-bound M^{2+} was often referred to as a stronger reductant, it is suspected that much of these M^{2+} was not accessible by nitrite in the case of Fe and ZnO NP. That is, a more passive oxide coating of varying thickness may always cover the solid/liquid interface and insulate NO_2^- from contacting the active metallic sites. Hence, lower adsorption efficiencies were observed in the case of Fe and ZnO NP when compared to Ag NP (Fig. 4). Moreover, the adsorption of nitrite on ZnO NP follows pseudo second-order kinetic and hence chemisorptions may occur in this case in terms of adsorbate electron affinity as well as local electrostatic attractions between the surface and adsorbate. Chemisorption on oxide surfaces is in many ways markedly different from chemisorption on metals. Oxide surfaces tend to exhibit acid/base or electron donor/acceptor interactions with adsorbates (Miletic et al. 2003).

In each nitrite adsorption/reduction experiment described here, nitrogen and ammonium are the only two products after complete adsorption. At low pH, as in the present investigation, the nitrogen is the desired product, whereas ammonium is the undesired one.

Conclusions

In the medium-term, the removal of nitrite from drinking water is necessary in order to protect the

environment and human health. In this work, Ag, Fe and ZnO NP prepared by EEW technique were effective adsorbents for the removal of nitrite from water with very high adsorption capacities at equilibrium. The suitability of the kinetic models for the adsorption of nitrite on the nanoparticles was investigated. Adsorption kinetic data were analyzed using the pseudo first-order, pseudo second-order and intraparticle diffusion model. Ag, Fe and ZnO NP have adsorption properties due to their novel structure, high surface area and high sorption capacities.

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Abdullah Alqudami was born in Hajjah, Yemen in 1976. In 1999 he graduated with honors from Physics Department of Sana'a University, Yemen. In 1999, he was appointed as junior academic staff at the Physics Department of Ibb University, Yemen and he got dual MSc scholarships granted by Indian Council for Cultural Relations (ICCR) and Ibb University to pursue MSc study at Jawaharlal Nehru University, New Delhi, India. In 2003, he received the M.Sc.

degree in Physics from JNU. He was granted a PhD scholarship from Ibb University and he joined the Lab of Professor S. Annapoorni of the Department of Physics & Astrophysics, University of Delhi, India, for four years of research in nanomaterials and he received the Ph.D. degree in November 2007. In January 2008, he joined back Ibb University, Yemen, and he have been appointed as an Assistant Professor of Nano Materials at the Physics Department. In September 2008, he was appointed as the Head of the Physics Department and remained so until September 2012.

As a researcher, he was awarded the President of the Republic of Yemen Award for the promotion of scientific research, first period 2009 for his project on water pollution treatment using metal nanoparticles. He has a good and well known research records in International Journals with good impact factors and good citations. His current and ongoing research includes the preparations, characterizations, and applications of metal and metal oxide nanoparticles. He is trying to control the preparation of such nanoparticles using electro-exploding wire technique, investigate their optical properties, use them for environmental applications such as water treatment, antibacterial, UV blocking, etc., and to explore their toxicity to animals and their biological effects.

Dr. Alqudami is a member of some local and international societies, a referee/reviewer for some international journals in the field of nanoscience and nanomaterials, a member of the editorial board of the *International Journal of Materials Science*, a member of the Scientific and Technical Committees & Editorial Review Boards of the World Academy of Science, a full member of the American Nano Society, and an Associate Member of the Institute of Nanotechnology, UK.